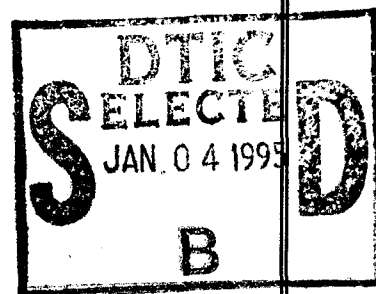


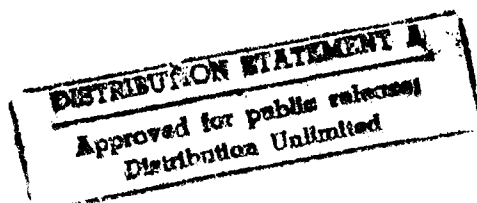
Quarterly Technical Report

Growth, Characterization and Device Development in Monocrystalline Diamond Films

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13. ABSTRACT (Maximum 200 words) Bias-enhanced nucleation (BEN) has been employed with TiC(111) substrates for the deposition of oriented diamond particles. The orientation of all the particles was the same, as observed via SEM. Some particles showed evidence of azimuthal twist and tilt, most likely due to the ~18% lattice mismatch with the TiC. Raman spectra exhibited a strong feature at 1332 cm ⁻¹ , indicative of diamond, and smaller features at 1480 cm ⁻¹ and 1602 cm ⁻¹ due to sp ² -bonded carbon. An increase in the duration of the BEN step and a reduction in the twinning are expected to increase the density of oriented particles. An optical analysis of the strain fields of point and line defects, and the distributions of these defects as well as the distribution of the optical centers in the diamond films has also been conducted. The nitrogen optical centers were uniformly distributed. Calculations indicated that line-type defects are far more detrimental to the stress than point defects.				
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I. Introduction

Diamond as a semiconductor in high-frequency, high-power transistors has unique advantages and disadvantages. Two advantages of diamond over other semiconductors used for these devices are its high thermal conductivity and high electric-field breakdown. The high thermal conductivity allows for higher power dissipation over similar devices made in Si or GaAs, and the higher electric field breakdown makes possible the production of substantially higher power, higher frequency devices than can be made with other commonly-used semiconductors.

In general, the use of bulk crystals severely limits the potential semiconductor applications of diamond. Among several problems typical for this approach are the difficulty of doping the bulk crystals, device integration problems, high cost and low area of such substrates. In principal, these problems can be alleviated via the availability of chemically vapor deposited (CVD) diamond films. Recent studies have shown that CVD diamond films have thermally activated conductivity with activation energies similar to crystalline diamonds with comparable doping levels. Acceptor doping via the gas phase is also possible during activated CVD growth by the addition of diborane to the primary gas stream.

The recently developed activated CVD methods have made feasible the growth of polycrystalline diamond thin films on many non-diamond substrates and the growth of single crystal thin films on diamond substrates. More specifically, single crystal epitaxial films have been grown on the {100} faces of natural and high pressure/high temperature synthetic crystals. Crystallographic perfection of these homoepitaxial films is comparable to that of natural diamond crystals. However, routes to the achievement of rapid nucleation on foreign substrates and heteroepitaxy on one or more of these substrates has proven more difficult to achieve. This area of study has been a principal focus of the research of this contract.

At present, the feasibility of diamond electronics has been demonstrated with several simple experimental devices, while the development of a true diamond-based semiconductor materials technology has several barriers which a host of investigators are struggling to surmount. It is in this latter regime of investigation that the research described in this report has and continues to address.

In this reporting period, bias-enhanced nucleation has been employed as a pretreatment process in tandem with TiC(111) substrates to explore the potential for growing epitaxial diamond films. This process enhanced the diamond nucleation density on this material. These results agree with previous research which showed that carbide-forming substrates are important for enhanced nucleation of diamond. Preferred orientation of the diamond(111) particles along the $\langle 110 \rangle$ of the substrates was observed. The strain fields of selected point and line defects, as well as the distributions of these defects and of the optical centers in diamond films has also been determined. Nitrogen point defects and their associated optical

centers were found to be uniformly distributed. The main sources of strain in the samples were line defects; the strain due to point defects was minimal.

The following subsections detail the experimental procedures for each of the aforementioned studies, discuss the results and provide conclusions and references for these studies. Note that each major section is self-contained with its own figures, tables and references.

II. Bias-enhanced Nucleation of Highly Oriented Diamond on TiC(111)

A. Introduction

Diamond has received much attention for use in a wide variety of applications. These include tribological, thermal management, optical, and microelectronic applications. In order to exploit this material's extreme properties, high quality diamond crystals must be formed. And in some cases, through process advancements, the properties of synthetic diamond have surpassed those exhibited by natural diamond.[1]

Diamond utilized for microelectronic application such as a semiconductor device is desirable but must contend with other materials possessing similar attributes, such as SiC. Both diamond and SiC exhibit high temperature semiconducting properties allowing for higher current densities, which may be used for higher power or smaller-scale circuitry applications. SiC, however, has undergone much more development and very large bulk crystals of this material may now be formed.

In regards to diamond deposition, epitaxial diamond has been achieved on both cBN and diamond,[2, 3] but these materials are expensive and large area substrates of these materials are difficult to obtain. There has been past research to suggest that diamond heteroepitaxy may be achieved on non-diamond or related substrates, however, the recent deposition on β -SiC, Si, and Ni have shown the most dramatic results.[4-7] In the case of the work conducted on β -SiC and Si, bias-enhanced nucleation (BEN) was used for the formation of these epitaxial diamond crystals.[8, 9] The term highly oriented diamond (HOD) has been used to describe these aligned particles rather than epitaxial diamond since once these particles coalesce, they have been observed to form low angle grain boundaries on the order of 6° to 10° . [8] The origin of this misalignment in the form of particle tilting or azimuthal misorientation is a result of an approximately 20% mismatch at the diamond/SiC interface. Zhu *et al.*[10] have shown that the misfit dislocations that form at the interface account for the misorientation.

It has been revealed through continued research of BEN that the carbide forming nature of the substrate appears to be an important attribute for the effective utilization of this nucleation pretreatment process.[11] Later work, on a study of several refractory metal substrates, indicated that a correlation was observed for both the induction time to the onset of significant diamond nucleation and the nucleation density to material properties exhibited by these metals.[12] It was proposed that this class of material would be likely candidates for further heteroepitaxy studies using BEN.

This letter discusses early results using BEN on single crystal TiC(111) substrates. Also, it was speculated in previous work that the refractory materials may be potential diamond heteroepitaxy substrates when using this unique diamond nucleation pretreatment.[11] In

general, the refractory metals are similar to silicon in the respect that they are carbide formers but quite different in other respects. One of these differences is that TiC, like the other refractory monocarbides, possess a different crystal structure to that of SiC (TiC-rock salt structure; SiC-zinc blende structure). Epitaxial diamond was formed on β -SiC and Si when utilizing this diamond nucleation pretreatment[4] and served as a guide for this research. Similar experimental conditions to the work performed by Stoner *et al.*[9] on β -SiC was used on TiC(111) to investigate the possibility of obtaining diamond heteroepitaxy using BEN.

B. Experimental Approach

Sample Preparation. The substrate material used in these experiments was TiC(111) which was obtained from Advanced Technology Materials, Inc. The single crystal material was cut into $10\text{mm} \times 5\text{mm} \times 1\text{mm}$ pieces and was polished using 30, 6, 1, and $0.1\mu\text{m}$ diamond grit followed by 1 hour of polishing using $0.05\mu\text{m}$ Al_2O_3 to remove any residual diamond that may have been embedded in the TiC surface. The substrates were subsequently cleaned in acetone, methanol, and isopropanol. Prior to entering into the chemical vapor deposition (CVD) reactor, a de-ionized water rinse was performed.

Experimental Conditions. The deposition system which was used in this study was an ASTeX 2.45 MHz, 1.5kW system that has been described in detail elsewhere.[13, 14] The samples were initially cleaned *in situ* in a H_2 plasma for 30 minutes to remove any oxygen present on the surface followed immediately by the BEN process for approximately 10 minutes. Following the *in situ* H_2 plasma cleaning procedure and BEN, the process parameters for yielding well faceted diamond particles were employed. The specific system parameters for each stage of the diamond deposition are listed in Table I.

Table I. System parameters for each process.

System parameter	H_2 plasma clean	BEN	Growth
Power (W)	600	600	600
Pressure (Torr)	25	15	40
$\text{CH}_4:\text{H}_2$ ratio	—	5%	0.2%
Bias current (mA)	—	120	—
Bias voltage (Vdc)	—	225 ± 10	—
Temperature ($^\circ\text{C}$)	660 ± 20	785 ± 20	900
Duration	30 min	10-15 min	8 hours

C. Results and Discussion

It was observed that the diamond nucleation density was significantly enhanced which supports previous work regarding diamond nucleation via bias enhanced nucleation on carbide

forming substrates.[11] Micrographs (Figs. 1 and 2) taken throughout the substrate showed (111) textured diamond particles having the same orientation to one another. However, a dense region of diamond particles oriented to each other could not be found. Since these particles were oriented to each other across the substrate and to pyramidal etch pits in the TiC(111) surface, it is assumed that these particles are highly oriented with respect to the substrate. These etch pits were observed after the polishing and cleaning procedures.

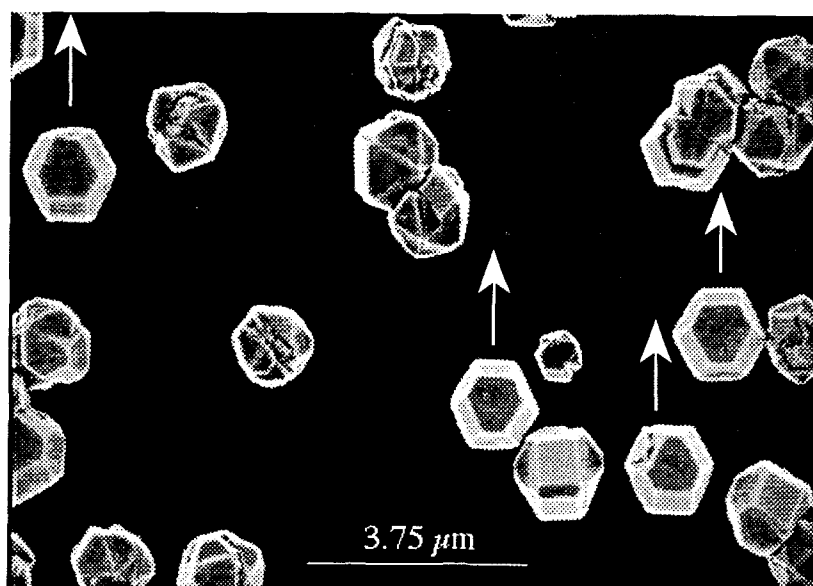


Figure 1. SEM micrograph of oriented diamond particles on the TiC (111) substrate with arrows showing orientation direction. The marker is 3.75μm in length.

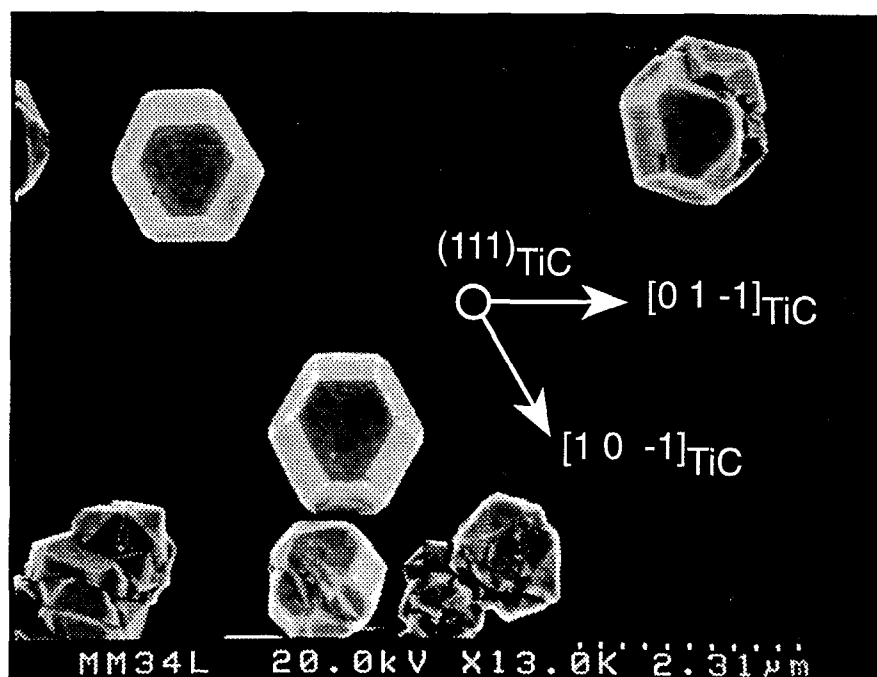


Figure 2. SEM micrograph of oriented diamond particles showing orientation of the TiC(111) substrate

The absence of a dense region with oriented diamond particles may be due the short BEN duration used. A moderate to low nucleation density was preferred to observed diamond particles separated from one another and to prevent coalescence into a complete film. Since the coefficient of thermal expansion are significantly different for TiC and diamond, delamination would have occurred upon the formation of a complete film. Also, the deposition conditions used for the growth of well faceted diamond crystals were not optimized and twinning on the diamond (111) face resulted. Therefore, a short BEN duration and growth conditions not favorable for suppressing twinning on the (111) face resulted in the low density of oriented diamond particles. The Raman spectra of the diamond particles in shown in Fig. 3 and indicates the characteristic diamond feature at 1332 cm^{-1} . The 1480 cm^{-1} and 1602 cm^{-1} features are indicative of sp^2 -bonded carbon and may be a result of the large occurrence of twinning.

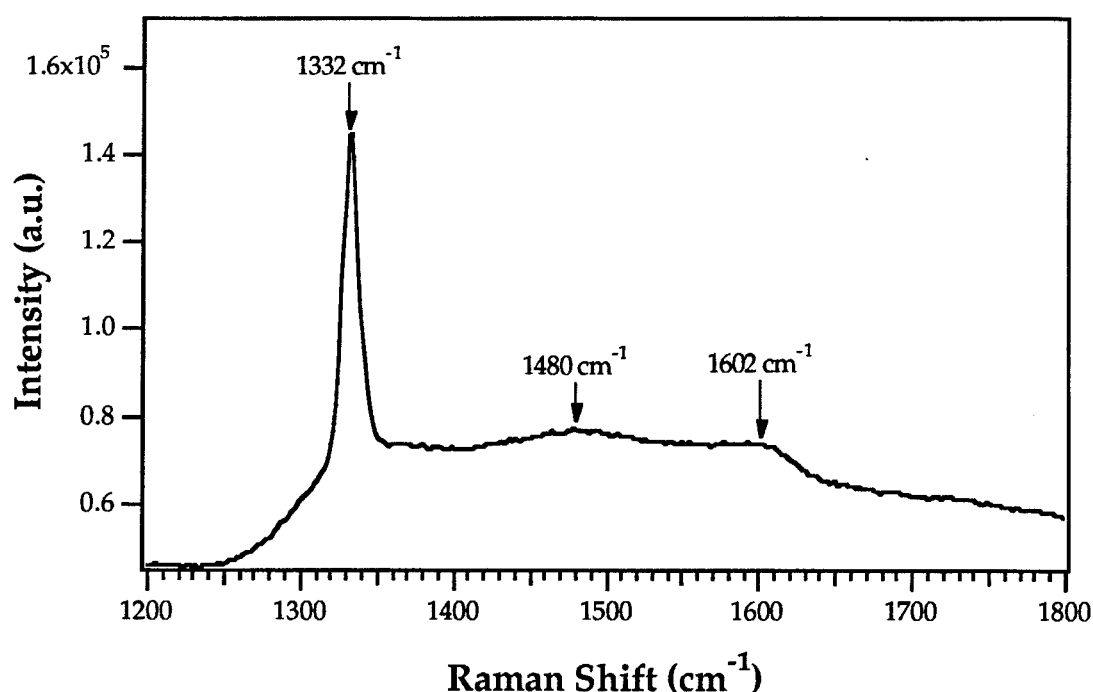


Figure 3. Micro-Raman spectrum of the diamond particles on the TiC(111) substrate. The spectrum shows a sharp peak at 1332 cm^{-1} characteristic of diamond and the features at 1480 cm^{-1} and 1602 cm^{-1} are characteristic of sp^2 -bonded carbon.

D. Conclusions

Bias enhanced nucleation was utilized on TiC(111) single crystal substrates in order to explore the possibility of obtaining epitaxial diamond. This nucleation pretreatment process was observed to enhance the diamond nucleation density on this material. This supports previous work in this area claiming that the carbide forming nature of the substrate is important in order to obtain enhanced nucleation of diamond. A preferred orientation of diamond (111)

textured particles was observed on the TiC(111) substrate. These particles were observed to be aligned with the substrate in the $\langle 110 \rangle$ direction.

E. Acknowledgments

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III. PL Lineshape Analysis and Stress Analysis in Diamond Films

A. Introduction

This section presents the initial results of an investigation of the PL lineshape of the two diamond films: combustion and HF 0.2%. The well-established theoretical work developed by Stoneham was applied on the strain broadening mechanisms of luminescence bands [1].

In a real crystal, the PL lineshape is determined almost entirely by the crystal inhomogeneities. In general, the inhomogeneous broadening is at least 1000 times greater than the homogeneous broadening, the latter which is due to the life time shortening of a transition. The principle mechanism of the inhomogeneous broadening at low temperature is the strain broadening that arises from the presence of dislocation type defects and point defects in the crystal. These defects introduce strain fields throughout the crystal that interact with and perturb the optical transitions energies. The statistical distribution of optical centers and defects in the crystal as well as their densities determine the variations in the transition energies of the optical centers. The resulting lineshape of the interaction of the strain with the optical center is schematically presented in Fig. 1.

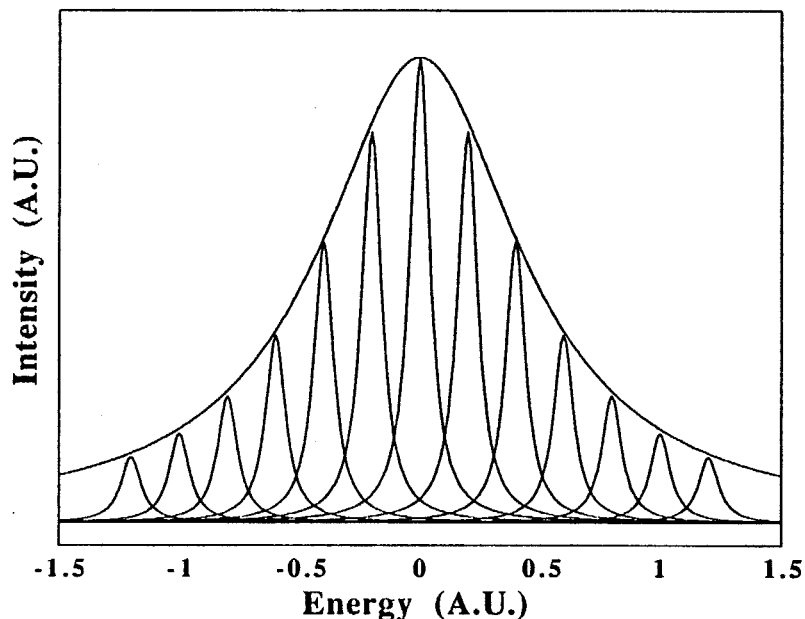


Figure 1. Inhomogeneous broadening.

In the following paragraph, a brief summary of Stoneham's main results about strain broadened luminescence bands is presented.

- (a) For a symmetric lattice in which the lattice sites accommodate at random the optical centers and defects, the luminescence lineshape is expected to be symmetric.

- (b) When the strain in the crystal arises solely from point defects of strength M and with perturbation strain field $e \sim M/r^3$ (where M is a measure of size mismatch) and the distribution of the optical centers and of the defects is uniform, the luminescence lineshape is expected to be a Lorentzian. The linewidth of a luminescence band is proportional to the density and to the strength of the point defects causing the strain, and also depends on the properties of the optical center in question (i.e., the response of the center to stress which is usually structure-dependent).
- (c) When the sources of the strain in the crystal are dislocations with perturbation strain field of the form $e \sim b/r$ (where b is the magnitude of the Burgers vector) and the dislocation type defects, as well as the optical centers are uniformly distributed in the crystal, the lineshape is expected to be a Gaussian. The linewidth of a luminescence band, in this case, is proportional to the square root of the dislocation density and to the magnitude of the Burgers vector, and also depends on the structure of the optical center.
- (d) In the case when both types of defects are present in the crystal, the resulting line is the convoluted lineshape of the Gaussian and the Lorentzian: this line is known as the Voigt profile and has no closed-form mathematical expression. The relative linewidths of the Gaussian component and the Lorentzian component in the Voigt profile reflect which defect is dominant in the crystal. For example, if there exists a very high concentration of dislocations relative to the concentration of point defects, the lineshape is expected to be more of a Gaussian shape.

The above results of Stoneham assumed an ideal continuous single crystal for which the spatial distribution of the defects and of the optical centers were taken to be uniform. Experimental evidence supporting Stoneham's work has been found which correlates the lineshape of the luminescence to the type, the concentration and the distribution of the defects. The above optical analysis of crystal stress and strain has been proven to be very useful, particularly in the cases when an appreciable amount of point defects as well as dislocations are present in the crystal.

In addition to the impurity type defects, a high concentration of line defects of dislocation type (attributed to the non equilibrium nature of the growth process) were found in diamond films similar to ours utilizing TEM spectroscopy. Hence, an optical analysis as proposed by Stoneham may be useful in order to differentiate the contributions to the strain from each defect type. Since the diamond films, which have grains and substrate interfaces and growth-biased incorporation of defects and optical centers, are not in the category of the ideal crystals assumed in Stoneham's work, it is expected that some of the PL lineshape to be influenced by the deviation from the ideal.

In this section, the results of an investigation of the 2.154 eV nitrogen optical band are presented. This PL band appeared in the PL spectra of our HF 0.2% which was intentionally

nitrogen doped, and in the spectra of our undoped combustion samples. The unintentional nitrogen doping of the combustion film was due to the relatively high level of nitrogen in the growth chamber. It was found that in both samples the nitrogen optical centers are uniformly distributed. The main sources of strain in the HF 0.2%N and in the combustion samples are due to line defects, the strain due to point defects were found to be minimal. The combustion film exhibits the lower value of strain.

B. The 2.154 eV Nitrogen-vacancy Optical Center

The 2.154 eV nitrogen optical center was suggested to consist of one nitrogen atom and a vacancy (or vacancies). This center appears in nitrogen-free diamonds after nitrogen implantation and annealing, or in diamonds containing nitrogen (Ia,Ib) after radiation damage and annealing. The conclusion that this center contains a single nitrogen atom was inferred from the observed linear dependence of the luminescence intensity on the implantation dose. The conclusion that this optical center contains, in addition to the nitrogen, a vacancy or vacancies was inferred from the following observation (supported by annealing experiment). In general, an optical center that contains vacancies (soft center) has a stronger response to strains and stresses than optical centers which do not contain vacancies (rigid centers). Hence, the luminescence linewidth of a soft center is expected to be relatively wider than the linewidth of a rigid center under the same stress state. Upon comparing the 2.154 eV PL linewidth with other known PL bands in the same diamond crystal, Collins *et al.* [2], found that the 2.154 eV linewidth was substantially wider.

Since the nitrogen atoms in diamond may appear in various configurations and forms (only some of which are optical centers), it is important to know which one of the nitrogen forms is the predominant source of the strain. According to Davies [3], at least 95% of the total nitrogen atoms in type I diamonds appear in the form of the small aggregate (which are considered as point defects) and this form has been established to be the main source of the strain. One very common form of small aggregate consists of two nearest-neighbor nitrogen atoms which is referred to as the A aggregate. While the A aggregate is not PL active and can be detected only via absorption spectroscopy, the presence of the A aggregate in our diamonds has been indirectly inferred from the existence of the PL band-A in the spectra. Therefore, we may safely assume that the A aggregate in our diamonds are the cause of most of the nitrogen related strain. Much smaller contribution is expected from the nitrogen optical centers themselves (like the 1.945 and 2.154 eV); hence we do not regard these optical centers as defects.

Figures 2 and 3 show the 2.154 eV PL bands of the HF 0.2%N and the combustion films respectively, as well as their Lorentzian and Gaussian lineshape (of the same widths as the PL linewidths). It can be seen from the figures that both PL lineshapes exhibit a high degree of symmetry. In this respect our PL bands are similar to the symmetric 2.154 eV PL lineshape of

la crystalline diamond [3]. The observed symmetry of our PL bands, applying Stoneham's theory, imply that the 2.154 eV optical centers as well as the defects causing the strain are uniformly distributed throughout the diamond films.

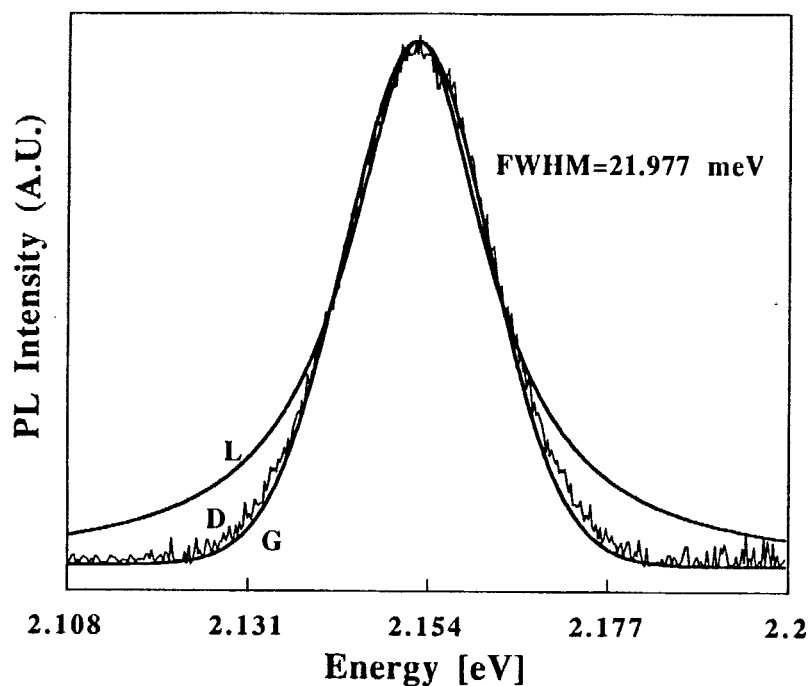


Figure 2. HF 0.2% n diamond, the 2.154 eV nitrogen center.

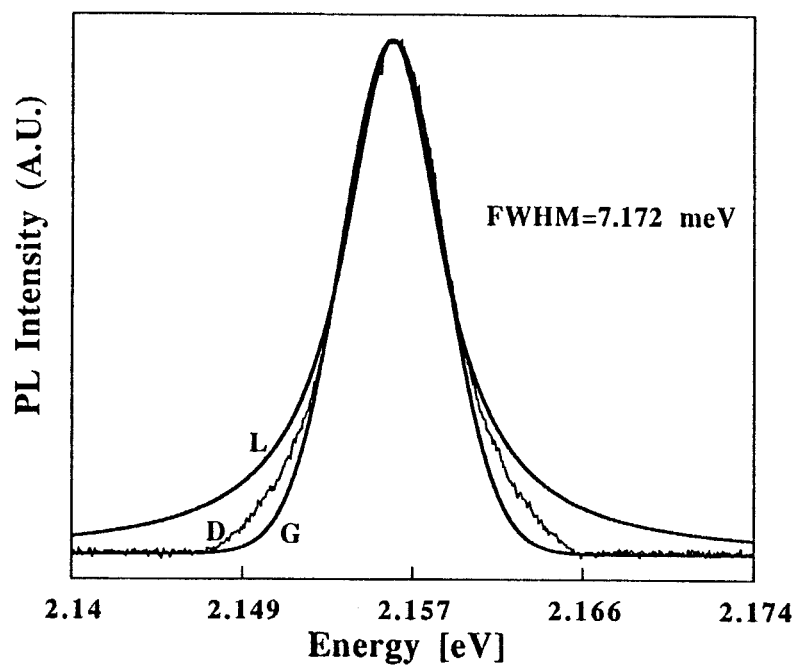


Figure 3. Combustion diamond, 2.154 eV center.

It can be seen in Figs. 2 and 3 that the PL lineshape of the HF 0.2% n film is mainly a Gaussian with a very small Lorentzian component: the linewidth of this PL band (Voit profile) is 21.977 meV. The lineshape of the combustion film lies between a Lorentzian and a Gaussian function, and thus constitutes a Voit profile of linewidth 7.172 meV. Since the relative linewidths of the Gaussian and Lorentzian components of each of the PL bands can be used to determine their respective stress contributions, it is imperative to calculate the linewidth values before proceeding to analyze the type of defects as related to the observed lineshapes.

It has been demonstrated that the Voit profile can be approximated by a linear combination of the Gaussian and the Lorentzian functions:

$$I(\omega)/I_0 = [1-W_L/W_V] \exp[-2.772\{(\omega-\omega_0)/W_V\}^2] + [W_L/W_V] \{1/(1+4((\omega-\omega_0)/W_V)^2)\} \quad (1)$$

where W_L is the Lorentzian linewidth (to be found) and W_V is the Voit linewidth (the known width of a given PL line). Once W_L has been determined from the curve fit of the given PL line to Eq. 1, the following relation between the Lorentzian and Gaussian linewidths:

$$W_V = W_L / 2 + \sqrt{W_L^2 / 4 + W_G^2} \quad (2)$$

is used to find the Gaussian linewidth component W_G .

Figure 4 shows that the PL line of the combustion film (obtained from high resolution PL spectroscopy) can be approximated well by Eq. 1; from this curve fit and Equation 2 the values of W_L and W_G are calculated to be 2.232 and 5.952 meV respectively. The ratio of $W_L/W_G=0.37$ implies that the Gaussian stress is approximately 2.7 times bigger than the Lorentzian stress.

Also, it has been demonstrated that the total stress, S , in a given diamond sample may be obtained from the linewidth W of a nitrogen PL via the following approximation :

$$S=W/10 \quad (3)$$

where S is in units of GPa and the linewidth W is in meV. Hence the stress of the combustion sample as obtained from its PL linewidth is ~0.72 GPa. Combining this result with the previously calculated ratio $W_L/W_G=0.37$, we find that the stress S_L which causes the Lorentzian broadening is ~0.19 GPa and the Gaussian stress S_G is ~0.53 GPa. The same analytical procedure was performed on the PL band of the HF 0.2% n. Table I summarizes the above results for both samples.

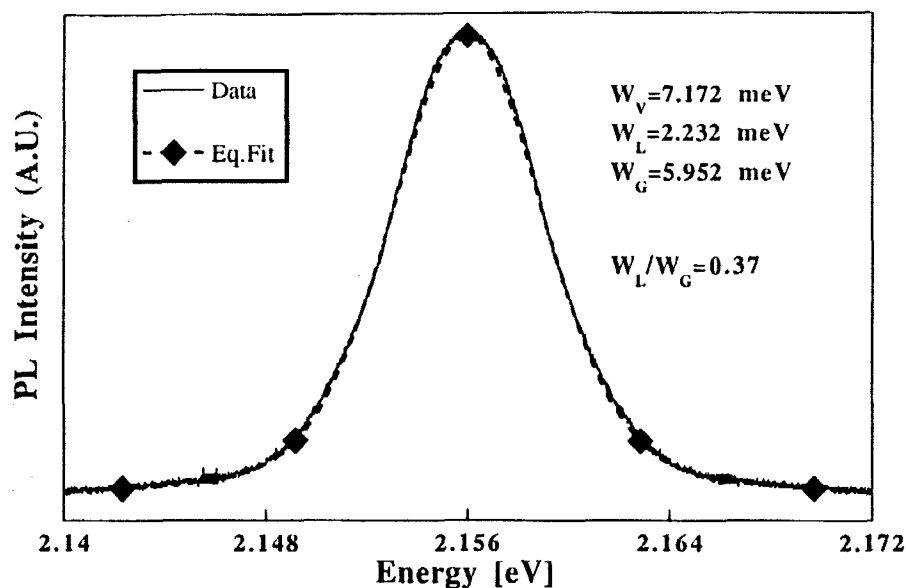


Figure 4. Combustion diamond, 2.154 eV nitrogen-vacancy center.

Table I. Summary of Results of Samples

Sample	Total stress S [GPa]	S_G [GPa]	S_L [GPa]
Combustion	0.72	0.53	0.19
HF 0.2%n	2.2	1.83	0.37

According to the results listed in Table I, we conclude that the HF 0.2%n and the combustion samples exhibit mainly a Gaussian Stress S_G and a much smaller Lorentzian stress S_L . Upon applying Stoneham's results, which were discussed in the introduction, to the above findings we may conclude that line-type defects are far more detrimental to the stress than point defects.

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